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PATENT SPECIFICATION

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NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements relating to the Cold-Working of Metals

We, THE PYRENE COMPANY LIMITED, a British Company, of Great West Road, Brentford, Middlesex, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

In order to facilitate the cold-working of metals, for example intube-drawing or wire-drawing, inorganic coatings of various kinds may be applied to the surface of the metal. For example phosphate coatings may be formed on the surface of iron, steel, aluminium or zinc. When lubricants are applied to such coatings they adhere better than to the surface of the bare metal, and thus facilitate cold-working. After cold-working a metal having on its surface such a coating, however, the appearance of the metal tends not to be bright and metallic, but light grey, dark grey or greenish, depending on the coating applied, and further treatment, involving several steps, for example grease-removal and treatment in acid or alkali may be necessary to obtain a bright surface. There is therefore a need for a lubricating coating which facilitates cold-working in the same way as such inorganic chemical coatings, without seriously impairing the appearance of the surface.

We have found that a very effective lubricating coating may be formed on a metal surface prior to cold-working by treating it with a metal-free solution or dispersion containing one or more cellulose ethers. The coating when dry should preferably contain at least 20% by weight of cellulose ether, particularly good results being obtained when it contains at least 35% by weight. By "metal-free" solutions or dispersions, we mean solutions or dispersions of cellulose ethers in which there is no elemental metal present as opposed to dissolved metal ions, and we also define "metal-free" solutions or dispersions accord-

ing to the present invention to mean that the solution or dispersion does not contain any molybdenum disulphide.

Of the cellulose ethers, methyl, carboxymethyl, ethyl, methylethyl, hydroxyethyl, ethylhydroxyethyl, and benzyl cellulose have proved to be particularly suitable. Of these, carboxymethyl cellulose, as its sodium salt, is soluble in water, methyl cellulose and hydroxyethyl cellulose are soluble both in cold water and in certain organic solvents, while the remainder dissolve only in certain organic solvents.

The following are examples of suitable organic solvents: chlorinated hydrocarbons, such as methylene chloride, tri- and perchloroethylene, alcohols, esters, ketones, and aromatic hydrocarbons. Non-inflammable solvents comprising one or more chlorinated hydrocarbons are particularly suitable, and where a solvent mixture is used it preferably contains sufficient chlorinated hydrocarbon to render it non-inflammable. A mixture of organic solvents can be used, these being generally preferred to water, since they are much more readily evaporated.

In addition to one or more cellulose ethers, the solution or dispersion from which the lubricating coating is obtained may contain one or more other ingredients, such as polychloroprene, a plasticiser, e.g. dioctyl sebacate and trichloroethyl phosphate, an alkyd resin modified with a fatty acid, polybutene, polyethylene, natural and/or synthetic waxes, fatty acids, e.g. stearic acid and coconut oil fatty acid, a fatty acid alkanolamide, e.g. coconut oil fatty acid monoethanolamide, an animal oil, an animal fat, a vegetable oil, and/or a vegetable fat, e.g. tallow, wool fat and rapeseed oil, and a polyvinyl alkyl ether, e.g. polyvinyl isobutyl ether. The use of one or more of these further ingredients can increase the drawing capacity of the coat-

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ing. A number of these additives, such as a polyvinyl alkyl ether, a plasticiser, a fatty acid or an alkyd resin modified with a fatty acid, also increase the tenacity with which the cellulose ether coating adheres to the metallic surface.

The solids content of the solution or dispersion is preferably such as to yield a dry coating weight of from 1 to 15 grams per square metre. In general thinner coatings do not facilitate drawing sufficiently, while thicker coatings tend not to lead to any improvement in the drawing capacity. On the contrary, they may have a disadvantageous effect caused by accumulation of the coating ingredients in the drawing tool. The precise concentration of the ingredients in the solvent used depends on the way in which the solution or dispersion is applied to the metallic surface, and also on its viscosity. The necessary concentration can be established quite simply by preliminary tests.

Pigments, e.g. graphite, talc, chalk and kaolin, may also be incorporated in the solution or dispersion of cellulose ether in order to increase the film strength of the coating produced. These additives should, however, only be used in the case of difficult working operations, since they make it more difficult to remove the coating after the cold-working process, and it may then be necessary to use a more drastic cleaning operation to produce a clean metal surface.

The solution or dispersion of a cellulose ether is conveniently applied to the metal surface at room temperature. The metal surface is preferably first cleaned so that it is free of grease, dust and corrosion products, and may then be treated with the solution or dispersion by conventional lacquering methods, e.g. by immersion, flooding, dipping and spraying. Depending upon the solvent used, the wet film produced may be dried at room temperature or at an elevated temperature. The coated metallic surface may then be worked; if desired however, under certain circumstances, a conventional lubricant such as mineral oil may also be used. For example, deep-drawn metal parts are more readily removed from a die when a mineral oil is also used.

The process of this invention is applicable to a wide number of metals, but is particularly valuable when it is desired to work difficult materials such as refined steel, chrome-nickel alloys, titanium and zirconium, and also where the cold-working operation itself is difficult, such as tube-drawing in which there is a considerable reduction in dimensions, cold impact-extrusion and deep-draw-

ing. The coating and any additional lubricant present after cold-working may conveniently be removed by washing the worked metal surface in an organic solvent, or by treatment with a solvent vapour. Such a cleaning operation is very much simpler than those necessary for removing phosphate coatings for example. The surfaces cleaned in this way are distinguished by their outstanding cleanliness.

A further advantage of the present process is that if organic solvents only are used, the production of undesirable aqueous effluents is avoided, thus eliminating what might otherwise be a serious disposal problem.

Coatings of synthetic resins have hitherto been applied to metal surfaces to facilitate their cold-working, but the coatings obtained in the present process were found to be surprisingly superior to those obtained with polystyrene, polyvinylchloride, an acrylic resin, polyvinylbutyral, polyvinylacetate, cellulose acetate, chlorinated rubber, a cumarone resin, a phenol-aldehyde resin and a melamine-aldehyde resin, as the following results show. A coating of each of these resins and of each of three cellulose ethers was formed on steel tubes, the coatings being applied from organic solvents. In all cases the weight of the dry coating obtained was approximately 10 grams/square metre. After evaporation of the solvent the steel tubes were drawn at a speed of 7 m/min. in accordance with the following programme: 20 mm. x 2 mm. → 17 mm. x 1.7 mm. → 14 mm. x 1.5 mm. → 11 mm. x 1.4 mm. → 9 mm. x 1.2 mm. → 8 mm. x 1 mm. → 7 mm. x 0.8 mm. → 6 mm. x 0.7 mm. (in each case, the first number denotes the external diameter, the second number the wall thickness). The results obtained are shown in the following Table.

TABLE I

Coating material	Number of draws possible before scoring or cracking of the tube occurred
Methyl cellulose	6
Ethylhydroxyethyl cellulose	7
Ethyl cellulose	5
Polystyrene	2
Polyvinyl chloride	2
Acrylic resin	2
Polyvinyl butyral	1
Polyvinyl acetate	1
Cellulose acetate	2
Chlorinated rubber	2
Coumarone resin	3
Phenol-aldehyde resin	2
Melamine-aldehyde resin	2

It will be seen that the coatings of cellulose ether permitted much more extensive cold-working of the steel tubes.

5 The following Table illustrates the results obtained using various ingredients in addition to a cellulose ether. The ingredients were dissolved or dispersed in a solution of ethyl cellulose in a mixture of equal volumes of 10 methylene chloride and perchloroethylene.

Bright steel tubes were immersed in the solutions or dispersions obtained, and after removal from them were dried in the atmosphere, the weight of the dry coatings obtained lying between 6 and 12 grams per square metre. The same drawing programme as above was then carried out, the following results being obtained:

TABLE 2

Solution composition (solvent: methylene chloride + perchloroethylene)		
Ethyl cellulose (g./L.)	Additive (g./L.)	Number of draws possible before scoring or cracking of the tube occurred
60	—	5
60	12 — alkyd resin modified with fatty acid	7
60	12 — trichloro-ethyl phosphate	7
60	12 — polybutene	7
60	12 — polyethylene ^{+) molecular weight 2500}	
60	12 — dioctyl sebacate	7
30	30 — polyvinyl isobutyl ether	7
30	35 — polychloroprene	7
60	12 — stearic acid	7
60	12 — coconut oil fatty acid	7

^{+) Dispersed in the solution}

5 Although soft chattering was audible in the first three cases, this had no bearing on the results obtained, although it is naturally desirable that the cold-working should proceed completely silently if possible.

10 Where the metal in question or the cold-working operation is a difficult one, it may be desirable to include phosphoric acid or an ester thereof to cause the formation of a phosphate-coating, thus increasing the adhesion of the coating to the metal surface.

When this is necessary, however, the sodium salt of carboxymethyl cellulose should not be used as it is incompatible with phosphate. Phosphoric acid esters derived from fatty alcohols having a carbon chain of 8 to 20 carbon atoms per molecule are particularly suitable esters. The following Table compares the results obtained with and without phosphoric acid. Steel tubes were coated as described above to yield a coating of about 10 grams per square metre.

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TABLE 3

Solution	Number of draws possible before scoring or cracking of the tube occurred
60 g./l. ethyl cellulose + 12 g./l. dioctyl sebacate in (1 vol. methylene chloride + 1 vol. perchloroethylene)	7
60 g./l. ethyl cellulose + 12 g./l. dioctyl sebacate + 30 g./l. phosphoric acid (85%) in (2 vols. methylene chloride + 2 vols. perchloroethylene + 1 vol. isopropanol)	8

WHAT WE CLAIM IS:—

5 1. A process in which a lubricating coating is formed on a metal surface by the application of a metal-free solution or dispersion containing one or more cellulose ethers to the surface and in which the metal is subsequently subjected to cold-working.

10 2. A process according to claim 1 in which the coating when dry contains at least 20% by weight of cellulose ether.

15 3. A process according to claim 2 in which the coating when dry contains at least 35% by weight of cellulose ether.

20 4. A process according to any one of claims 1 to 3 in which the coating has a dry film weight of from 1 to 15 grams per square metre.

25 5. A process according to any one of the preceding claims in which the coating is formed by the application of a solution or dispersion of one or more cellulose ethers and one or more of the following:—polychloroprene, polyethylene, polybutene, a natural wax, a synthetic wax, an alkyd resin modified with a fatty acid, a plasticiser, a fatty acid, a fatty acid alkanolamide, an animal oil, an animal fat, a vegetable oil, a vegetable fat, a polyvinyl alkyl ether, a pigment.

30 6. A process according to any one of the preceding claims in which the coating is formed by application of a solution in a volatile organic solvent.

7. A process according to claim 6 in which the organic solvent is non-inflammable and comprises one or more chlorinated hydrocarbons. 35

8. A process according to any one of the preceding claims in which there is used one or more of the following cellulose ethers:—methyl, carboxymethyl, ethyl, methylethyl, hydroxyethyl, ethylhydroxyethyl, and benzyl cellulose. 40

9. A process according to any one of the preceding claims in which phosphoric acid or an ester thereof is included in the solution or dispersion to form a phosphate coating. 45

10. A process according to any one of the preceding claims in which the lubricating coating is itself treated with a conventional lubricant prior to cold-working. 50

11. A process according to Claim 1 in which a lubricating coating containing a cellulose ether is formed on a metal surface and the metal is subjected to cold-working substantially as herein described with reference to any of the results given in the Tables herein. 55

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